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⑯ ISOMERIZATION OF WAXY LUBE OILS AND PETROLEUM WAXES USING A
SILICOALUMINOPHOSPHATE MOLECULAR SIEVE CATALYST.

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⑯ References cited:

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US-A- 4 440 871	US-A- 4 610 778
US-A- 4 650 917	US-A- 4 686 029
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Description

The present invention relates to a process for isomerizing a waxy feed, including petroleum waxes, over a catalyst comprising an intermediate pore size silicoaluminophosphate molecular sieve and at least one Group VIII metal to produce a lube oil having a low pour point and excellent viscosity and viscosity index properties.

High-quality lubricating oils are critical for the operation of modern machinery and automobiles. Unfortunately, the supply of natural crude oils having good lubricating properties is not adequate for present demands. Due to uncertainties in world crude oil supplies, high-quality lubricating oils must be produced from ordinary crude feedstocks. Numerous processes have been proposed for producing lubricating oils that can be converted into other products by upgrading the ordinary and low-quality stocks.

It is desirable to upgrade a crude fraction otherwise unsuitable for lubricant manufacture into one from which good yields of lube oils can be obtained. Dewaxing is required when highly paraffinic oils are to be used in products which need to remain mobile at low temperatures, e.g., lubricating oils, heating oils and jet fuels. The higher molecular weight straight chain normal and slightly branched paraffins which are present in oils of this kind are waxes which cause high pour points and high cloud points in the oils. If adequately low pour points are to be obtained, these waxes must be wholly or partly removed. In the past, various solvent removal techniques were used such as propane dewaxing and MEK dewaxing but these techniques are costly and time consuming. Catalytic dewaxing processes are more economical and achieve this end by selectively cracking the longer chain n-paraffins to produce lower molecular weight products, some of which may be removed by distillation.

Because of their selectivity, prior art dewaxing catalysts generally comprise an aluminosilicate zeolite having a pore size which admits the straight chain n-paraffins either alone or with only slightly branched chain paraffins, but which excludes more highly branched materials, cycloaliphatics and aromatics. Zeolites such as ZSM-5, ZSM-11, ZSM-12, ZSM-23, ZSM-35 and ZSM-38 have been proposed for this purpose in dewaxing processes and are described in U.S. Patent Nos. 3,894,938; 4,176,050; 4,181,598; 4,222,855; 4,229,282 and 4,247,388.

Since dewaxing processes of this kind function by means of cracking reactions, a number of useful products become degraded to lower molecular weight materials. For example, waxy paraffins may be cracked down to butane, propane, ethane and methane as may the lighter n-paraffins which do not contribute to the waxy nature of the oil. Because these lighter products are generally of lower value than the higher molecular weight materials, it would obviously be desirable to limit the degree of cracking which takes place during a catalytic dewaxing process.

Although EP-A-0 209 997 and U.S. Patent Nos. 3,700,585; 3,894,938; 4,176,050; 4,181,598; 4,222,855; 4,229,282 and 4,247,388 teach dewaxing of waxy feeds, the processes disclosed therein do not disclose a process for producing high yields of a lube oil having a very low pour point and a high viscosity index from feeds containing a very high wax content, i.e., greater than 80% wax, such as slack wax or deoiled wax.

Since processes which remove wax will give a low yield with very waxy feeds, isomerization processes are preferred. U.S. Patent No. 4,734,539 discloses a method for isomerizing a naphtha feed using a medium pore zeolite catalyst, such as an H-offretite catalyst. U.S. Patent No. 4,518,485 discloses a process for dewaxing a hydrocarbon feedstock containing paraffins by a hydrotreating and isomerization process.

U. S. Patent No. 4,689,138 discloses an isomerization process for reducing the normal paraffin content of a hydrocarbon oil feedstock using a catalyst comprising an intermediate pore size silicoaluminophosphate molecular sieve containing a Group VIII metal component which is occluded in the crystals during growth.

Lube oils may also be prepared from feeds having a high wax content such as slack wax by an isomerization process. In prior art wax isomerization processes, however, either the yield is low and thus the process is uneconomical, or the feed is not completely dewaxed. When the feed is not completely dewaxed it must be recycled to a dewaxing process, e.g. a solvent dewaxer, which limits the throughput and increases cost. U.S. Patent No. 4,547,283 discloses converting wax to lube. However, the MEK dewaxing following isomerization disclosed therein severely limits pour reduction and thus, very low pour points cannot be achieved. Further, the catalyst disclosed therein is much less selective than the silicoaluminophosphate catalyst used in the present invention.

The present invention overcomes the problems and disadvantages of the prior art by providing a process for preparing a lube oil comprising isomerizing a waxy feed over a catalyst comprising an intermediate pore size silicoaluminophosphate molecular sieve and at least one Group VIII metal.

It is an object of the invention to produce a lube oil having excellent viscosity and viscosity index properties and a very low pour point.

It is a further object of the invention to provide a lube oil having low frictional resistance.

It is an additional object of the invention to provide a lube oil which causes less engine wear than prior art mineral oils.

It is an additional object of the invention to provide a lube oil which provides increased fuel efficiency.

5 It is yet a further object of the invention to provide a lube oil having improved oxidation resistance compared to conventional mineral oils. The term "oxidation resistance" refers to the resistance of the oil to oxygen addition, i.e., how rapidly oxygen is picked up and added to molecular species within the oil. Oxidation resistance is indicated by the oxidator BN measured in hours. Oxidator BN measures the time required for 100 grams of oil to absorb one liter of oxygen and is described in U.S. Patent No. 3,852,207 at 10 column 6, lines 15-30.

The present invention provides a process for producing lube oil having a viscosity index greater than 120 and a pour point of less than -9.40° C (15° F), which process comprises dewaxing a waxy feed containing at least 50% of wax or at least 70% of paraffinic carbon by isomerizing the feed over a catalyst comprising an intermediate pore size silicoaluminophosphate molecular sieve and at least one Group VIII 15 metal at a pressure of from 0.21 MPa to 13.9 MPa (15 psig to 2000 psig).

15 The invention thus provides a process for producing a lube oil have excellent viscosity and viscosity index properties and a low pour point.

The lube oil produced by the process of the invention has a high paraffinic content which provides a low viscosity for its boiling range. Such properties result in less frictional resistance, less engine wear and increased fuel efficiency. The low viscosity and low pour point of the lube oil result in easier cold engine 20 starting than prior art mineral oils. The lube oil has a high viscosity index ranging from about 120 to about 180 and a very low pour point, ranging from less than -80° F (-61.6° C) to about 15° F (-9.4° C). Since little or no viscosity index improver compositions or pour point depressant compositions are necessary for use with the lube oil of the invention, fewer performance-decreasing deposits are formed. The high paraffinic 25 nature of the oil gives it improved oxidation resistance compared to conventional mineral oils.

Reference will be made to the drawings in which:

FIG. 1 is a ternary diagram showing the compositional parameters of the silicoaluminophosphates of U.S. Patent No. 4,440,871 in terms of mole fractions of silicon, aluminum and phosphorus;

30 FIG. 2 is a ternary diagram showing the preferred compositional parameters of the silicoaluminophosphates of mole fractions of silicon, aluminum and phosphorus;

FIG. 3 is a plot of 700° F + (371.1° C) lube yield versus pour point for the process set forth in Example 3; and

FIG. 4 is a plot of viscosity index versus pour point for the process set forth in Example 3.

Reference will now be made in detail to the present preferred embodiments of the invention.

35 As used herein, the term "waxy feed" includes petroleum waxes. The feedstock employed in the process of the invention contains greater than about 50% wax, more preferably greater than about 80% wax. However, a highly paraffinic feed having a high pour point but containing less than 25% wax is also suitable for use in the process of the invention. Such a feed should contain greater than 70% paraffinic carbon, more preferably greater than 80% paraffinic carbon, most preferably greater than 90% paraffinic 40 carbon.

Exemplary additional suitable feeds for use in the process of the invention include waxy distillate stocks such as gas oils, lubricating oil stocks, synthetic oils such as those made by Fischer-Tropsch synthesis, foots oils, slack waxes and deoiled waxes. Foots oil is prepared by separating oil from the wax. The isolated oil is referred to as foots oil.

45 Feeds also suitable for use the process of the invention are partially dewaxed oils wherein dewaxing to an intermediate pour point has been carried out by a process other than that claimed herein, for example, conventional catalytic dewaxing processes and solvent dewaxing processes. Exemplary suitable solvent dewaxing processes are set forth in U.S. Patent No. 4,547,287.

The process of the invention may also be employed in combination with conventional dewaxing 50 processes to achieve a lube oil having particular desired properties. For example, the process of the invention can be used to reduce the pour point of a lube oil to a desired degree. Further reduction of the pour point can then be achieved using a conventional dewaxing process. The pour point of the lube oil produced by the process of the invention can be reduced by adding pour point depressant compositions thereto.

55 The feedstock will normally be a C₂₀ + feedstock generally boiling above about 600° F (315.5° C). However, the process of the invention is particularly useful with waxy distillate stocks such as gas oils, lubricating oil stocks, heating oils and other distillate fractions whose pour point and viscosity need to be maintained within certain specification limits. Lubricating oil stocks will generally boil above 230° C (450° F),

more usually above 315°C (600°F). Hydroprocessed stocks are a convenient source of stocks of this kind and also of other distillate fractions since they normally contain significant amounts of waxy n-paraffins. The feedstock of the present process will normally be a C₂₀ + feedstock containing paraffins, olefins, naphthenes, aromatics and heterocyclic compounds and a substantial proportion of higher molecular weight n-paraffins and slightly branched paraffins which contribute to the waxy nature of the feedstock. During processing, the n-paraffins and the slightly branched paraffins undergo some cracking or hydrocracking to form liquid range materials which contribute to a low viscosity product. The degree of cracking which occurs is, however, limited so that the yield of low boiling products is reduced, thereby preserving the economic value of the feedstock.

Slack wax can be obtained from either a hydrocracked lube oil or a solvent refined lube oil. Hydrocracking is preferred because that process can also reduce the nitrogen content to low values. With slack wax derived from solvent refined oils, defining can be used to reduce the nitrogen content. Optionally, hydrotreating of the slack wax can be carried out to lower the nitrogen content thereof. Slack waxes possess a very high viscosity index, normally in the range of from 140 to 200, depending on the oil content and the starting material from which the wax has been prepared. Slack waxes are therefore eminently suitable for the preparation of lubricating oils having very high viscosity indices, i.e., from about 120 to about 180.

While the process of the invention can be practiced with utility when the feed contains organic nitrogen (nitrogen containing impurities), it is preferred that the organic nitrogen content of the feed be less than about 50 ppmw, more preferably less than about 10 ppmw. Particularly good results, in terms of activity and length of catalyst cycle (period between successive regenerations or start-up and first regeneration) are experienced when the feed contains less than about 10 ppmw of organic nitrogen.

The phrase "intermediate pore size", as used herein means an effective pore aperture in the range of from about 5.3 to about 6.5 Angstroms when the molecular sieve is in the calcined form. Molecular sieves having pore apertures in this range tend to have unique molecular sieving characteristics. Unlike small pore zeolites such as erionite and chabazite, they will allow hydrocarbons having some branching into the molecular sieve void spaces. Unlike larger pore zeolites such as the faujasites and mordenites, they can differentiate between n-alkanes and slightly branched alkanes, and larger branched alkanes having, for example, quaternary carbon atoms.

The effective pore size of the molecular sieves can be measured using standard adsorption techniques and hydrocarbonaceous compounds of known minimum kinetic diameters. See Breck, Zeolite Molecular Sieves, 1974 (especially Chapter 8); Anderson et al., J. Catalysis 58, 114 (1979); and U.S. Patent No. 4,440,871, the pertinent portions of which are incorporated herein by reference.

Intermediate pore size molecular sieves will typically admit molecules having kinetic diameters of 5.3 to 6.5 Angstroms with little hindrance. Examples of such compounds (and their kinetic diameters in Angstroms) are: n-hexane (4.3), 3-methylpentane (5.5), benzene (5.85), and toluene (5.8). Compounds having kinetic diameters of about 6 to 6.5 Angstroms can be admitted into the pores, depending on the particular sieve, but do not penetrate as quickly and in some cases are effectively excluded. Compounds having kinetic diameters in the range of 6 to 6.5 Angstroms include: cyclohexane (6.0), 2,3-dimethylbutane (6.1), and m-xylene (6.1). Generally, compounds having kinetic diameters of greater than about 6.5 Angstroms do not penetrate the pore apertures and thus are not absorbed into the interior of the molecular sieve lattice. Examples of such larger compounds include: o-xylene (6.8), 1,3,5-trimethylbenzene (7.5), and tributylamine (8.1).

The preferred effective pore size range is from about 5.5 to about 6.2 Angstroms.

In performing adsorption measurements to determine pore size, standard techniques are used. It is convenient to consider a particular molecule as excluded if it does not reach at least 95% of its equilibrium adsorption value on the molecular sieve in less than about 10 minutes (p/p₀ = 0.5; 25°C).

The catalyst used in the isomerization process of the invention has an acidic component and a platinum and/or palladium hydrogenation component. The acidic component preferably comprises an intermediate pore size silicoaluminophosphate molecular sieve which is described in U.S. Patent No. 4,440,871, the pertinent disclosure of which is incorporated herein by reference.

The most preferred intermediate pore size silicoaluminophosphate molecular sieve for use in the process of the invention is SAPO-11. SAPO-11 comprises a molecular framework of corner-sharing [SiO₄] tetrahedra, [AlO₄] tetrahedra and [PO₄] tetrahedra, [i.e., (Si_xAl_yP)O₄ tetrahedral units]. When combined with a platinum or palladium hydrogenation component, the SAPO-11 converts the waxy components to produce a lubricating oil having excellent yield, very low pour point, low viscosity and a high viscosity index.

SAPO-11 comprises a silicoaluminophosphate material having a three-dimensional microporous crystal framework structure of [PO₄], [AlO₄] and [SiO₄] tetrahedral units whose unit empirical formula on an

anhydrous basis is:



5 wherein "R" represents at least one organic templating agent present in the intracrystalline pore system; "m" represents the moles of "R" present per mole of $(Si_xAl_yP_z)O_2$ and has a value of from zero to about 0.3, "x", "y" and "z" represent respectively, the mole fractions of silicon, aluminum and phosphorus, said mole fractions being within the compositional area bounded by points A, B, C, D and E on the ternary diagram of FIG. 1 or preferably within the area bounded by points a, b, c, d and e on the ternary diagram of 10 FIG. 2. The silicoaluminophosphate has a characteristic X-ray powder diffraction pattern which contains at least the d-spacings (as-synthesized and calcined) set forth below in Table I. When SAPO-11 is in the as-synthesized form "m" preferably has a value of from 0.02 to 0.3.

TABLE I

15

20

	2θ	d	Relative Intensity
	9.4-9.65	9.41-9.17	m
	20.3-20.6	4.37-4.31	m
	21.0-21.3	4.23-4.17	vs
	22.1-22.35	4.02-3.99	m
	22.5-22.9 (doublet)	3.95-3.92	m
	23.15-23.35	3.84-3.81	m-s

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All of the as-synthesized SAPO-11 compositions for which X-ray powder diffraction data have been obtained to date have patterns which are within the generalized pattern of Table II below.

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TABLE II

	2θ	d	100 x I/I ₀
5	8.05-8.3	10.98-10.65	20-42
	9.4-9.65	9.41-9.17	36-58
	13.1-13.4	6.76-6.61	12-16
	15.6-15.85	5.68-5.59	23-38
	16.2-16.4	5.47-5.40	3-5
10	18.95-19.2	4.68-4.62	5-6
	20.3-20.6	4.37-4.31	36-49
	21.0-21.3	4.23-4.17	100
	22.1-22.35	4.02-3.99	47-59
	22.5-22.9 (doublet)	3.95-3.92	55-60
15	23.15-23.35	3.84-3.81	64-74
	24.5-24.9 (doublet)	3.63-3.58	7-10
	26.4-26.8 (doublet)	3.38-3.33	11-19
	27.2-27.3	3.28-3.27	0-1
	28.3-28.5 (shoulder)	3.15-3.13	11-17
20	28.6-28.85	3.121-3.094	
	29.0-29.2	3.079-3.058	0-3
	29.45-29.65	3.033-3.013	5-7
	31.45-31.7	2.846-2.823	7-9
	32.8-33.1	2.730-2.706	11-14
25	34.1-34.4	2.629-2.607	7-9
	35.7-36.0	2.515-2.495	0-3
	36.3-36.7	2.475-2.449	3-4
	37.5-38.0 (doublet)	2.398-2.368	10-13
	39.3-39.55	2.292-2.279	2-3
30	40.3	2.238	0-2
	42.2-42.4	2.141-2.132	0-2
	42.8-43.1	2.113-2.099	3-6
	44.8-45.2 (doublet)	2.023-2.006	3-5
	45.9-46.1	1.977-1.969	0-2
35	46.8-47.1	1.941-1.929	0-1
	48.7-49.0	1.870-1.859	2-3
	50.5-50.8	1.807-1.797	3-4
	54.6-54.8	1.681-1.675	2-3
	55.4-55.7	1.658-1.650	0-2

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Another intermediate pore size Silicoaluminophosphate molecular sieve preferably used in the process of the invention is SAPO-31. SAPO-31 comprises a silicoaluminophosphate having a three-dimensional microporous crystal framework of [PO₂]_n, [AlO₂]_n and [SiO₂]_n tetrahedral units whose unit empirical formula on an anhydrous basis is:

45



wherein R represents at least one organic templating agent present in the intracrystalline pore system; "m" represents the moles of "R" present per mole of (Si_xAl_yP_z)O₂ and has a value of from zero to 0.3; "x", "y" and "z" represent respectively, the mole fractions of silicon, aluminum and phosphorus, said mole fractions being within the compositional area bounded by points A, B, C, D and E on the ternary diagram of FIG. 1, or preferably within the area bounded by points a, b, c, d and e on the ternary diagram of FIG. 2. The Silicoaluminophosphate has a characteristic X-ray powder diffraction pattern (as-synthesized and calcined) which contains at least the d-spacings set forth below in Table III. When SAPO-31 is in the as-synthesized form, "m" preferably has a value of from 0.02 to 0.3.

TABLE III

	2θ	d	Relative Intensity
5	8.5-8.6	10.40-10.28	m-s
	20.2-20.3	4.40-4.37	m
	21.9-22.1	4.06-4.02	w-m
	22.6-22.7	3.93-3.92	vs
	31.7-31.8	2.823-2.814	w-m

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All of the as-synthesized SAPO-31 compositions for which X-ray powder diffraction data have presently been obtained have patterns which are within the generalized pattern of Table IV below.

TABLE IV

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	2θ	d	100 x I/I ₀
20	6.1	14.5	0-1
	8.5-8.6*	10.40-10.28	60-72
	9.5*	9.31	7-14
	13.2-13.3*	6.71-6.66	1-4
	14.7-14.8	6.03-5.99	1-2
25	15.7-15.8*	5.64-5.61	1-8
	17.05-17.1	5.20-5.19	2-4
	18.3-18.4	4.85-4.82	2-3
	20.2-20.3	4.40-4.37	44-55
	21.1-21.2*	4.21-4.19	6-28
30	21.9-22.1*	4.06-4.02	32-38
	22.6-22.7*	3.93-3.92	100
	23.3-23.35	3.818-3.810	2-20
	25.1*	3.548	3-4
	25.65-25.75	3.473-3.460	2-3
35	26.5*	3.363	1-4
	27.9-28.0	3.198-3.187	8-10
	28.7*	3.110	0-2
	29.7	3.008	4-5
	31.7-31.8	2.823-2.814	15-18
40	32.9-33.0*	2.722-2.714	0-3
	35.1-35.2	2.557-2.550	5-8
	36.0-36.1	2.495-2.488	1-2
	37.2	2.417	1-2
	37.9-38.1*	2.374-2.362	2-4
45	39.3	2.292	2-3
	43.0-43.1*	2.103-2.100	1
	44.8-45.2*	2.023-2.006	1
	46.6	1.949	1-2
	47.4-47.5	1.918	1
50	48.6-48.7	1.872-1.870	2
	50.7-50.8	1.801-1.797	1
	51.6-51.7	1.771-1.768	2-3
	55.4-55.5	1.658-1.656	1

* Possibly contains peak from a minor impurity.

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SAPO-41, also suitable for use in the process of the invention, comprises a silicoaluminophosphate having a three-dimensional microporous crystal framework structure of [PO₄], [AlO₄] and [SiO₄] tetrahedral units, and whose unit empirical formula on an anhydrous basis is:



wherein R represents at least one organic templating agent present in the intracrystalline pore system; "m" represents the moles of "R" present per mole of $(Si_xAl_yP_z)O_2$ and has a value of from zero to 0.3; "x", "y", and "z" represent respectively, the mole fractions of silicon, aluminum, and phosphorus, said mole fractions being within the compositional area bounded by points A, B, C, D and E on the ternary diagram of FIG. 1, or preferably within the area bounded by points a, b, c, d and e on the ternary diagram of FIG. 2, and said silicoaluminophosphate having a characteristic X-ray powder diffraction pattern (as-synthesized and calcined) which contains at least the d-spacings set forth below in Table V. When SAPO-41 is in the as-synthesized form "m" preferably has a value of from 0.02 to 0.3.

TABLE V

	2θ	d	Relative Intensity
15	13.6-13.8	6.51-6.42	w-m
	20.5-20.6	4.33-4.31	w-m
	21.1-21.3	4.21-4.17	vs
20	22.1-22.3	4.02-3.99	m-s
	22.8-23.0	3.90-3.86	m
	23.1-23.4	3.82-3.80	w-m
	25.5-25.9	3.493-3.44	w-m

25 All of the as-synthesized SAPO-41 compositions for which X-ray powder diffraction data have presently been obtained have patterns which are within the generalized pattern of Table VI below.

TABLE VI

	2θ	d	100 x I/I ₀
30	6.7-6.8	13.19-12.99	15-24
	9.6-9.7	9.21-9.11	12-25
	13.6-13.8	6.51-6.42	10-28
35	18.2-18.3	4.87-4.85	8-10
	20.5-20.6	4.33-4.31	10-32
	21.1-21.3	4.21-4.17	100
	22.1-22.3	4.02-3.99	45-82
40	22.8-23.0	3.90-3.87	43-58
	23.1-23.4	3.82-3.80	20-30
	25.2-25.5	3.53-3.49	8-20
	25.5-25.9	3.493-3.44	12-28
	29.3-29.5	3.048-3.028	17-23
45	31.4-31.6	2.849-2.831	5-10
	33.1-33.3	2.706-2.690	5-7
	37.6-37.9	2.392-2.374	10-15
	38.1-38.3	2.362-2.350	7-10
	39.6-39.8	2.276-2.265	2-5
50	42.8-43.0	2.113-2.103	5-8
	49.0-49.3	1.856-1.848	1-8
	51.5	1.774	0-8

The process of the invention may also be carried out using a catalyst comprising an intermediate pore size nonzeolitic molecular sieve containing AlO_2 and PO_2 tetrahedral oxide units, and at least one Group VIII metal. Exemplary suitable intermediate pore size nonzeolitic molecular sieves are set forth in European Patent Application No. 158,977.

The intermediate pore size molecular sieve is used in admixture with at least one Group VIII metal. Preferably the Group VIII metal is selected from the group consisting of at least one of platinum and

5 palladium and optionally, other catalytically active metals such as molybdenum, nickel, vanadium, cobalt, tungsten, zinc, and mixtures thereof. Most preferably, the Group VIII metal is selected from the group consisting of at least one of platinum and palladium. The amount of metal ranges from about 0.01% to about 10% by weight of the molecular sieve, preferably from about 0.2% to about 5% by weight of the molecular sieve. The techniques of introducing catalytically active metals into a molecular sieve are disclosed in the literature, and pre-existing metal incorporation techniques and treatment of the molecular sieve to form an active catalyst such as ion exchange, impregnation or occlusion during sieve preparation are suitable for use in the present process. Such techniques are disclosed in U.S. Patent Nos. 3,236,761; 3,226,339; 3,236,762; 3,620,960; 3,373,109; 4,202,996, 4,440,781 and 4,710,485.

10 The term "metal" or "active metal" as used herein means one or more metals in the elemental state or in some form such as sulfide, oxide and mixtures thereof. Regardless of the state in which the metallic component actually exists, the concentrations are computed as if they existed in the elemental state.

15 The physical form of the catalyst depends on the type of catalytic reactor being employed and may be in the form of a granule or powder, and is desirably compacted into a more readily usable form (e.g., larger agglomerates), usually with a silica or alumina binder for fluidized bed reaction, or pills, prills, spheres, extrudates, or other shapes of controlled size to accord adequate catalyst-reactant contact. The catalyst may be employed either as a fluidized catalyst, or in a fixed or moving bed, and in one or more reaction stages.

20 The catalytic isomerization step of the invention may be conducted by contacting the feed with a fixed stationary bed of catalyst, with a fixed fluidized bed, or with a transport bed. A simple and therefore preferred configuration is a trickle-bed operation in which the feed is allowed to trickle through a stationary fixed bed, preferably in the presence of hydrogen.

25 The catalytic isomerization conditions employed depend on the feed used and the desired pour point. Generally, the temperature is from about 200 °C to about 475 °C, preferably from about 250 °C and to about 450 °C. The pressure is from 0,21 MPa to 13,9 MPa (15 psig and to 2000 psig), preferably from 0,45 MPa to 7,0 MPa (50 to 1000 psig), more preferably from 0,79 MPa to 4,1 MPa (100 psig to 600 psig). The process of the invention is preferably carried out at low pressure. The liquid hourly space velocity (LHSV) is preferably from about 0.1 to about 20, more preferably from about 0.1 to about 5, and most preferably from about 0.1 to about 1.0. Low pressure and low liquid hourly space velocity provide enhanced isomerization 30 selectivity which results in more isomerization and less cracking of the feed thus producing an increased yield.

35 Hydrogen is preferably present in the reaction zone during the catalytic isomerization process. The hydrogen to feed ratio is typically from about 500 to about 30,000 SCF/bbl (standard cubic feet per barrel), preferably from about 1,000 to about 20,000 SCF/bbl. Generally, hydrogen will be separated from the product and recycled to the reaction zone.

40 The intermediate pore size molecular sieve used in the isomerization step provides selective conversion of the waxy components to non-waxy components. During processing, isomerization of the paraffins occurs to reduce the pour point of the oil below that of the feed and form lube oil boiling range materials which contribute to a low pour point product having excellent viscosity index properties.

45 Because of the selectivity of the intermediate pore size silicoaluminophosphate molecular sieve used in the invention, the yield of low boiling products is reduced, thereby preserving the economic value of the feedstock.

The intermediate pore size molecular sieve catalyst can be manufactured into a wide variety of physical forms. The molecular sieves can be in the form of a powder, a granule, or a molded product, such as an extrudate having a particle size sufficient to pass through a 2-mesh (Tyler) screen and be retained on a 40-mesh (Tyler) screen. In cases wherein the catalyst is molded, such as by extrusion with a binder, the silicoaluminophosphate can be extruded before drying, or, dried or partially dried and then extruded.

50 The molecular sieve can be composited with other materials resistant to temperatures and other conditions employed in the isomerization process. Such matrix materials include active and inactive materials and synthetic or naturally occurring zeolites as well as inorganic materials such as clays, silica and metal oxides. The latter may be either naturally occurring or in the form of gelatinous precipitates, gels or sols including mixtures of silica and metal oxides. Inactive materials suitably serve as diluents to control the amount of conversion in the isomerization process so that products can be obtained economically without employing other means for controlling the rate of reaction. The molecular sieve may be incorporated 55 into naturally occurring clays, e.g., bentonite and kaolin. These materials, i.e., clays, oxides, etc., function, in part, as binders for the catalyst. It is desirable to provide a catalyst having good crush strength because in petroleum refining, the catalyst is often subjected to rough handling. This tends to break the catalyst down into powder-like materials which cause problems in processing.

Naturally occurring clays which can be composited with the molecular sieve include the montmorillonite and kaolin families, which families include the sub-bentonites, and the kaolins commonly known as Dixie, McNamee, Georgia and Florida clays or others in which the main mineral constituent is halloysite, kaolinite, dickite, nacrite or anauxite. Fibrous clays such as halloysite, sepiolite and attapulgite can also be used as supports. Such clays can be used in the raw state as originally mined or initially subjected to calcination, acid treatment or chemical modification.

In addition to the foregoing materials, the molecular sieve can be composited with porous matrix materials and mixtures of matrix materials such as silica, alumina, titania, magnesia, silica-alumina, silica-magnesia, silica-zirconia, silica-thoria, silica-beryllia, silica-titania, titania-zirconia as well as ternary compositions such as silica-alumina-thoria, silica-alumina-titania, silica-alumina-magnesia and silica-magnesia-zirconia. The matrix can be in the form of a cogel.

The catalyst used in the process of this invention can also be composited with other zeolites such as synthetic and natural faujasites, (e.g., X and Y) erionites, and mordenites. It can also be composited with purely synthetic zeolites such as those of the ZSM series. The combination of zeolites can also be composited in a porous inorganic matrix.

It is often desirable to use mild hydrogenation referred to as hydrofinishing after isomerization to produce more stable lubricating oils. Hydrofinishing is typically conducted at temperatures ranging from about 190 °C to about 340 °C, at pressures from about 400 psig to about 3000 psig, at space velocities (LHSV) from about 0.1 to about 20, and hydrogen recycle rates of from about 400 to about 1500 SCF/bbl. The hydrogenation catalyst employed must be active enough not only to hydrogenate the olefins, diolefins and color bodies within the lube oil fractions, but also to reduce the aromatic content. The hydrofinishing step is beneficial in preparing an acceptably stable lubricating oil.

Suitable hydrogenation catalysts include conventional metallic hydrogenation catalysts, particularly the Group VIII metals such as cobalt, nickel, palladium and platinum. The metals are typically associated with carriers such as bauxite, alumina, silica gel, silica-alumina composites, and crystalline aluminosilicate zeolites. Palladium is a particularly preferred hydrogenation metal. If desired, non-noble Group VIII metals can be used with molybdates. Metal oxides or sulfides can be used. Suitable catalysts are disclosed in U.S. Patent Nos. 3,852,207; 4,157,294; 3,904,513 and 4,673,487.

The high viscosity index lube oil produced by the process of the present invention can be used as a blending component to raise the viscosity index of lube oils to a higher value. Since yield decreases with increasing viscosity index in either hydrocracking or solvent refining, the use of an isomerized wax to increase the viscosity index improves yield.

The invention will be further clarified by the following examples, which are intended to be purely exemplary of the invention.

The wax content of the oil set forth in the following Examples was determined as follows:

300 g of oil was diluted 50/50 with a 4:1 mixture of methyl ethyl ketone and toluene which was cooled to -20 °C in a refrigerator. The mixture was filtered through a Coors funnel at -15 °C using Whatman No. 3 filter paper. The wax was removed from the filter and placed in a tared 2 liter flask. The solvent was removed on a hot plate and the wax weighed.

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Example 1

SAPO-11 was prepared as follows:

472.4 g of 85% H₃PO₄ were added to 1208 g of distilled H₂O in a Teflon beaker in an ice bath. 816.8 g of aluminum isopropoxide (Al[OC₃H₇]₃) were slowly added with mixing and then mixed until homogenous. Then 120 g of fumed silica (Cabosil M-5) were added with mixing. Next, 182.4 g of di-n-propylamine (Pr₂NH) were added, followed by 9.0 g of 85% H₃PO₄. Final mixing was done using a Polytron. The mixture had a pH of 6.5 and the following composition, expressed in molar ratio of oxides:

50 0.9Pr₂NH:SiO₂:Al₂O₃:1.04P₂O₅:36H₂O

The mixture was placed in a Teflon bottle in a stainless steel pressure vessel and heated for 5 days at 200 °C with no stirring and autogenous pressure. The supernatant liquid was removed and the product was filtered, washed with water, dried overnight at 121 °C, and calcined in air for 8 hours at 566 °C. The average crystallite size was less than 0.5 micron. The product was identified as SAPO-11 by X-ray diffraction analysis. Elemental analysis of the calcined sieve showed it to have the following anhydrous molar composition:

0.37SiO₂:Al₂O₃:P₂O₅

The sieve was bound with 35% Catapal alumina and made into 1/10 inch extrudate. The extrudate was dried 3 hours at 121 °C, calcined in air for 4 hours at 454 °C, then impregnated with 0.5% Pt (as Pt(NH₃)-₆Cl₂·H₂O) by the pore-fill method. It was then dried for 2 hours at 93 °C, 2 hours at 149 °C, and calcined in air for 8 hours at 454 °C. For the following examples, the catalyst was crushed and screened to 24-42 mesh.

Example 2

The catalyst of Example 1 was used to isomerize a 800 °F + (426.6 °C) hydrocracked waxy vacuum gas oil (Table VII) at 15,18 MPa (2200 psig), 1 LHSV, and 8 M SCF/bbl once-through H₂. The catalyst was also run at 2.9 MPa (400 psig). The results are set forth in Table VIII and demonstrate that high lube yield at very low pour point is achieved by the present process. The data also evidences the substantial yield benefit from operating at a lower reactor pressure. Lube yield is defined as

$$\frac{g(\text{product})}{g(\text{feed})} \times 100\%$$

where the product has the same 5 LV% boiling point as the feed.

Example 3

The catalyst of Example 1 was used to isomerize a slack wax derived from a 900 °F + (482.2 °C) hydrocracked vacuum gas oil (Table IX). Conditions were 1.1 MPa (160 psig) reactor pressure, 0.3 LHSV, and 8 M SCF/bbl once-through H₂. At a catalyst temperature of 680 °F (356.4 °C), a -27 °C pour point oil of 7.1 cSt viscosity at 100 °C and 149 viscosity index was produced. Pour point reduction could be increased by raising the catalyst temperature. Figure 3 shows a plot of 700 °F + lube yield versus pour point, where 700 °F + lube yield is defined as:

$$1 - \frac{g_{700^{\circ}\text{F}+}(371.1^{\circ}\text{C}) \text{ (feed)} - g_{700^{\circ}\text{F}+}(371.1^{\circ}\text{C}) \text{ (product)}}{g_{700^{\circ}\text{F}+}(371.1^{\circ}\text{C}) \text{ (feed)}} \times 100\%$$

Figure 4 shows a plot of viscosity index versus pour point.

Example 4

The catalyst of Example 1 was used to isomerize a deoiled slack wax derived from a 700-800 °F (371.1 - 426.6 °C) solvent refined vacuum gas oil (Table X). Conditions were 1.1 MPa (160 psig), 0.3 LHSV, and 8 M SCF/bbl once-through H₂. The results are set forth in Table XI.

Example 5

The catalyst of Example 1 was used to isomerize a deoiled slack wax derived from a 800-900 °F (426.6 - 482.2 °C) solvent refined vacuum gas oil (Table XII). Conditions were the same as in Example 4. The results are set forth in Table XIII.

Example 6

A -30 °C pour point oil prepared in Example 3 was subsequently hydrofinished over a 0.5% Pd on silica-alumina catalyst at 500 °F (260.0 °C), 15,18 MPa (2200 psig), 0.25 LHSV, and 8 M SCF/bbl H₂. Inspections of the hydrofinished oil are given in Table XIV.

TABLE VII

800 °F + (426.6 °C) Hydrocracked Waxy VGO		
5	Gravity, °API	40.0
	Wax, Wt.%	25.0
	Pour Point, °C	+39
	Viscosity at 100 °C, cSt	4.585
	VI	152
10	Simulated Distillation, LV%, °F	
	ST/5	749/769 (394.4/405.4 °C)
	10/30	778/803 (410.3/424.1 °C)
	50	826 (436.7 °C)
15	70/90	857/920 (453.8/488.4 °C)
	95/99	956/1016 (508.2/541.2 °C)
	Hydrocarbon Distribution, LV%	
20	Paraffins	54
	Naphthenes	29
	Aromatics	17
	Carbon Type Distribution, ndM, (D3238-85), wt%	
25	Paraffinic	92
	Naphthenic	8
	Aromatic	0

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TABLE VIII
Isomerizing 800°F+ (426.6°C) Hydrocracked Waxy
VGO at 1 LHSV and 8 M SCR/bbl H₂

Pressure, psig	Catalyst Temperature, °F	Yield, wt%	Pour/Cloud, °C	Viscosity at 40/100°C, cSt	VI
2000 (13.9 MPa)	670 (350.9°C)	74.5	-12/-6	18.87/4.305	139
	690 (361.9°C)	63.3	-24/-19	19.08/4.301	136
	710 (372.9°C)	44.9	-48/-47	19.56/4.294	129
400 (2.9 MPa)	650 (343.3°C)	69.7	-45/-28	19.43/4.349	116
	670 (350.9°C)	55.0	<-63/<-63	19.64/4.304	128

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TABLE IX

Slack Wax from 900 °F + (482.2 °C +) Hydrocracked VGO			
5	Gravity, °API		23.2
	Sulfur, ppm		2.2
	Nitrogen, ppm		< 1
10	Pour Point, °C		+63
	Viscosity, cSt,	100 °C	8.30
		70 °C	16.55
15	Oil in Wax, Wt%		14.5
	Simulated Distillation, LV%, °F		
20	ST/5		802/860 (423.5/455.4 °C)
	10/30		885/933 (469.2/495.6 °C)
	50		960 (510.4 °C)
	70/90		995/1038 (529.7/553.3 °C)
	95/99		1063/1118 (567.1/597.3 °C)

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TABLE X

Decoiled Slack Wax from 700-800 °F (371.1 - 426.6 °C) Solvent Refined VGO		
5	Gravity, °API	41.0
	Pour Point, °C	+51
	Viscosity, cSt, 100 °C	3.141
	Sulfur, ppm	383
	Nitrogen, ppm	8.9
	N-paraffins, Wt.%	77.2
Simulated Distillation, LV%, °F		
10	ST/5	617/674 (321.75/353.1 °C)
	10/30	695/732 (364.65/385 °C)
	50	760 (400.4 °C)
	70/90	795/842 (419.7/445.5 °C)
	95/99	861/903 (456/479.1 °C)

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TABLE XI

Catalyst Temperature °F	650°F+ Yield, wt. %	Pour/Cloud, °C	Viscosity at 40/100°C, cSt	VI
640 (334.4°C)	72.0	-18/-5	9.563/2.817	151
650 (340°C)	77.1	-15/-2	9.883/2.899	154
655 (342.7°C)	81.1	-15/0	10.10/2.918	150

TABLE XII

Deoiled Slack Wax from 800-900 °F (426.6/482.2 °C) Solvent Refined VGO			
5	Gravity, °API		40.2
	Pour Point, °C		+57
10	Viscosity, cSt, 100 °C		4.317
	70 °C		7.554
15	Sulfur, ppm		122
	Nitrogen, ppm		3.4
	N-paraffins, Wt.%		57.1
20	Simulated Distillation, LV%, °F		
	ST/5		565/714 (293.2/375.1 °C)
	10/30		738/808 (388.3/426.8 °C)
	50		843 (446.1 °C)
	70/90		873/909 (462.6/482.4 °C)
	95/99		928/965 (492.8/513.2 °C)

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TABLE XIII

Solvent Refined Oil at 1,1 MPa (160 psig), 0.3 NSV, and 8 N SCF/mol H₂

Isomerizing Dotted Slack Wax From 800-900°F (426.6-482.2°C)

Catalyst Temperature, °F	700°F (371.1°C) Yield, Wt. %	Pour/Cloud, °C	Viscosity at 40/100°C, cSt	VI
655 (342.7°C)	90.4	+ 6/+17	15.44/4.017	169
670 (350.9°C)	85.3	- 9/+3	15.59/3.938	155
685 (359.2°C)	79.5	-24/-6	16.16/3.997	152
700 (371.1°C)	77.4	-27/-9	16.73/4.050	147

TABLE XIV

Hydrofinished Oil from Example 6		
5	Color	< 1.0
	Gravity, °API	37.0
	Aniline Pt, °F	259.6
10	Pour Point, °C	-30
	Cloud Point, °C	-7
	Sulfur, ppm	0.4
	Nitrogen, ppm	< 1
15	Viscosity, cSt	
	40 °C	39.33
	100 °C	7.073
	VI	143
	Oxidator BN, hr	40.9
20	Carbon Type Distribution, ndM, Wt.%	
	Paraffinic	80.27
	Naphthenic	19.73
	Aromatic	0.00
25	Simulated Distillation, LV%, °F	
	ST/5	630/702 (328.9/368.5 °C)
	10/30	749/862 (394.4/456.5 °C)
	50	916 (486.2 °C)
	70/90	957/1005 (508.8 °C)
30	95/99	1030/1084 (548.9/578.6 °C)

Claims

1. A process for producing lube oil having a viscosity index greater than 120 and a pour point of less than -9.40 °C (15 °F), which process comprises dewaxing a waxy feed containing at least 50% of wax or at least 70% of paraffinic carbon by isomerizing the feed over a catalyst comprising an intermediate pore size silicoaluminophosphate molecular sieve and at least one Group VIII metal at a pressure of from 0.21 MPa to 13.9 MPa (15 psig to 2000 psig).
2. A process according to claim 1 wherein said waxy feed contains at least 80% wax.
3. A process according to claim 1 wherein said waxy feed contains at least 90% paraffinic carbon.
4. A process according to any of claims 1 to 3 wherein said waxy feed, containing at least 50% of wax or at least 70% of paraffinic carbon, is selected from gas oil, lubricating oil stock, synthetic oil, foots oil, slack wax and deoiled wax.
5. A process according to any of claims 1 to 4 wherein said silicoaluminophosphate molecular sieve is selected from SAPO-11, SAPO-31 and SAPO-41 and said metal compound is selected from at least one of platinum and palladium.
6. A process according to any of claims 1 to 4 wherein said silicoaluminophosphate molecular sieve is a SAPO-11 crystalline silicoaluminophosphate.
7. A process according to claim 7 wherein said silicoaluminophosphate molecular sieve component further comprises a platinum hydrogenation component.

8. A process according to any of claims 1 to 7 wherein the isomerization is carried out at a temperature of from 200° C to 475° C.
9. A process according to claim 10 wherein the isomerization is carried out at a pressure of from 0.45 MPa to 7.0 MPa (50 to 1000 psig).
10. A process according to claim 8 wherein isomerization is carried out at a temperature from 250° C to 450° C and at a pressure of from 0.79 MPa to 4.1 MPa (100 psig to 600 psig).
11. A process according to any of claims 1 to 10 wherein the liquid hourly space velocity during isomerization is from 0.1 to 20.
12. A process according to claim 11 wherein the liquid hourly space velocity is from 0.1 to 5.
13. A process according to any of claims 1 to 12 wherein isomerization is carried out in the presence of hydrogen.
14. A process according to any of claims 1 to 13 further comprising hydrofinishing the isomerized lube oil.
15. A process according to claim 14 wherein the hydrofinishing is carried out at a temperature of from 190° C to 340° C and a pressure of from 2.9 MPa to 20.8 MPa (400 psig to 3000 psig).
16. A process according to claim 14 to 15 wherein hydrofinishing is carried out in the presence of a metallic hydrogenated catalyst.
17. A process according to any of claims 1 to 16 wherein said waxy feed has an organic nitrogen content of less than 50 ppmw.
18. A process according to claim 17 wherein said organic nitrogen content is less than 10 ppmw.
19. A process for producing lube oil having a viscosity index greater than 120 and a pour point of less than -9.4° C (15° F), which process comprises dewaxing a waxy feed containing at least 50% of wax or at least 70% of paraffinic carbon by isomerizing a waxy feed over a catalyst comprising (1) an intermediate pore size nonzeolitic molecular sieve containing AlO₂ and PO₂ tetrahedral oxide units and (2) at least one Group VIII metal at a pressure of from 0.21 MPa to 13.9 MPa (15 psig to 2000 psig).
20. A process according to claim 19 wherein said waxy feed, containing at least 50% of wax or at least 70% of paraffinic carbons, is selected from gas oil, lubricating oil stock, synthetic oil, foots oil, slack wax and deoiled wax.
21. A process according to claim 19 or 20 wherein the liquid hourly space velocity during isomerization is from 0.1 to 20.
22. A process according to any of claims 19 to 21 further comprising hydrofinishing the isomerized lube oil.
23. Lube oil prepared according to anyone of claims 1 to 22, having a viscosity index of at least 140 and a pour point no greater than -12° C.
24. Lube oil prepared according to anyone of claims 1 to 22, having a viscosity index of at least 120 and a pour point no greater than -40° C.

Patentansprüche

1. Verfahren zur Herstellung von Schmieröl mit einem Viskositätsindex größer als 120 und einem Fließpunkt von weniger als -9,40° C (15° F), bei dem eine wachshaltige Beschickung, die mindestens 50 % Wachs oder mindestens 70 % Paraffinkohlenstoff enthält, entwachst wird durch Isomerisierung der Beschickung über einem Katalysator, der ein Silikoaluminophosphat-Molekularsieb mittlerer Porengröße und mindestens ein Metall der Gruppe VIII enthält, bei einem Druck von 0,21 MPa bis 13,9 MPa

(15 psig bis 2000 psig).

2. Verfahren nach Anspruch 1, bei dem die wachshaltige Beschickung mindestens 80 % Wachs enthält.
- 5 3. Verfahren nach Anspruch 1, bei dem die wachshaltige Beschickung mindestens 90 % Paraffinkohlenstoff enthält.
- 10 4. Verfahren nach einem der Ansprüche 1 bis 3, bei dem die wachshaltige Beschickung, die mindestens 50 % Wachs oder mindestens 70 % Paraffinkohlenstoff enthält, aus Gasöl, Schmieröldestillatmaterial, synthetischem Öl, Schwitzöl, Paraffingatsch oder entöltem Wachs besteht.
- 15 5. Verfahren nach einem der Ansprüche 1 bis 4, bei dem das Silikoaluminophosphat-Molekularsieb aus SAPO-11, SAPO-31 oder SAPO-41 und die Metallverbindung aus mindestens einer solchen von Platin oder Palladium bestehen.
- 20 6. Verfahren nach einem der Ansprüche 1 bis 4, bei dem das Silikoaluminophosphat-Molekularsieb aus SAPO-11 vom Typ kristallines Silikoaluminophosphat besteht.
7. Verfahren nach Anspruch 6, bei dem die Silikoaluminophosphat-Molekularsiebkomponente ferner eine Platin-Hydrierkomponente enthält.
- 25 8. Verfahren nach einem der Ansprüche 1 bis 7, bei dem die Isomerisierung bei einer Temperatur von 200 ° C bis 475 ° C durchgeführt wird.
9. Verfahren nach Anspruch 10, bei dem die Isomerisierung bei einem Druck von 0,45 MPa bis 7,0 MPa (50 psig bis 1000 psig) durchgeführt wird.
- 30 10. Verfahren nach Anspruch 8, bei dem die Isomerisierung bei einer Temperatur von 250 ° C bis 450 ° C und bei einem Druck von 0,79 MPa bis 4,1 MPa (100 psig bis 600 psig) durchgeführt wird.
11. Verfahren nach einem der Ansprüche 1 bis 10, bei dem die stündliche Flüssigkeitsraumgeschwindigkeit während der Isomerisierung von 0,1 bis 20 beträgt.
- 35 12. Verfahren nach Anspruch 11, bei dem die stündliche Flüssigkeitsraumgeschwindigkeit von 0,1 bis 5 beträgt.
13. Verfahren nach einem der Ansprüche 1 bis 12, bei dem die Isomerisierung in Gegenwart von Wasserstoff durchgeführt wird.
- 40 14. Verfahren nach einem der Ansprüche 1 bis 13, das ferner eine Hydrofinish-Behandlung des isomérisierten Schmieröls umfaßt.
15. Verfahren nach Anspruch 14, bei dem die Hydrofinish-Behandlung bei einer Temperatur von 190 ° C bis 340 ° C und bei einem Druck von 2,9 MPa bis 20,8 MPa (400 psig bis 3000 psig) durchgeführt wird.
- 45 16. Verfahren nach den Ansprüchen 14 bis 15, bei dem die Hydrofinish-Behandlung in Gegenwart eines Metall-Hydrierkatalysators durchgeführt wird.
17. Verfahren nach einem der Ansprüche 1 bis 16, bei dem die wachshaltige Beschickung einen organischen Stickstoffgehalt von weniger als 50 ppmw aufweist.
- 50 18. Verfahren nach Anspruch 17, bei dem der organische Stickstoffgehalt weniger als 10 ppmw beträgt.
19. Verfahren zur Herstellung von Schmieröl mit einem Viskositätsindex größer als 120 und einem Fließpunkt von weniger als -9,4 ° C (15 ° F), bei dem eine wachshaltige Beschickung, die mindestens 50 % Wachs oder mindestens 70 % Paraffinkohlenstoff enthält, entwachst wird durch Isomerisierung einer wachshaltigen Beschickung über einem Katalysator, der (1) ein nichtzeolithisches Molekularsieb mittlerer Porengröße mit AlO₂- und PO₂-tetraedrischen Oxideinheiten und (2) mindestens ein Metall der

Gruppe VIII enthält, bei einem Druck von 0,21 MPa bis 13,9 MPa (15 psig bis 2000 psig).

20. Verfahren nach Anspruch 19, bei dem die wachshaltige Beschickung, die mindestens 50 % Wachs oder mindestens 70 % Paraffinkohlenstoff enthält, aus Gasöl, Schmieröldestillatmaterial, synthetischem Öl, Schwitzöl, Paraffingatsch oder entöltem Wachs besteht.
- 5 21. Verfahren nach den Ansprüchen 19 oder 20, bei dem die stündliche Flüssigkeitsraumgeschwindigkeit während der Isomerisierung von 0,1 bis 20 beträgt.
- 10 22. Verfahren nach einem der Ansprüche 19 bis 21, bei dem das isomerisierte Schmieröl ferner einer Hydrofinish-Behandlung unterworfen wird.
- 15 23. Schmieröl, hergestellt nach einem der Ansprüche 1 bis 22, mit einem Viskositätsindex von mindestens 140 und einem Fließpunkt von nicht größer als -12° C.
24. Schmieröl, hergestellt nach einem der Ansprüche 1 bis 22, mit einem Viskositätsindex von mindestens 120 und einem Fließpunkt von nicht größer als -40° C.

Revendications

20. 1. Procédé de production d'une huile lubrifiante ayant un indice de viscosité supérieur à 120 et un point d'écoulement inférieur à -9,40 °C (15 °F), procédé qui comprend le déparaffinage d'une charge cireuse contenant au moins 50 % de paraffines, ou au moins 70 % de carbone paraffinique par isomérisation de la charge sur un catalyseur comprenant un tamis moléculaire consistant en un silicoaluminophosphate à diamètre intermédiaire des pores et au moins un métal du Groupe VIII sous une pression de 0,21 MPa à 13,9 MPa (15 psig à 2000 psig).
- 25 2. Procédé suivant la revendication 1, dans lequel la charge paraffinique contient au moins 80 % de paraffines.
- 30 3. Procédé suivant la revendication 1, dans lequel la charge paraffinique contient au moins 90 % de carbone paraffinique.
- 35 4. Procédé suivant l'une quelconque des revendications 1 à 3, dans lequel la charge paraffinique, contenant au moins 50 % de paraffines ou au moins 70 % de carbone paraffinique, est choisi entre un gasoil, une huile lubrifiante de base, une huile synthétique, une huile de ressusage, une paraffine non déshuilée et une paraffine déshuilée.
- 40 5. Procédé suivant l'une quelconque des revendications 1 à 4, dans lequel le tamis moléculaire consistant en un silicoaluminophosphate est choisi entre le SAPO-11, le SAPO-31 et le SAPO-41, et le composé métallique consiste en au moins un composé choisi entre un composé de platine et un composé de palladium.
- 45 6. Procédé suivant l'une quelconque des revendications 1 à 4, dans lequel le tamis moléculaire consistant en un silicoaluminophosphate est un silicoaluminophosphate cristallin SAPO-11.
7. Procédé suivant la revendication 7, dans lequel le tamis moléculaire consistant en un silicoaluminophosphate comprend en outre un constituant d'hydrogénéation à base de platine.
- 50 8. Procédé suivant l'une quelconque des revendications 1 à 7, dans lequel l'isomérisation est effectuée à une température de 200 °C à 475 °C.
9. Procédé suivant la revendication 10, dans lequel l'isomérisation est effectuée sous une pression de 0,45 MPa à 7,0 MPa (50 à 1000 psig).
- 55 10. Procédé suivant la revendication 8, dans lequel l'isomérisation est effectuée à une température de 250 °C à 450 °C et sous une pression de 0,79 MPa à 4,1 MPa (100 psig à 600 psig).

11. Procédé suivant l'une quelconque des revendications 1 à 10, dans lequel la vitesse spatiale horaire de liquide au cours de l'isomérisation est comprise dans l'intervalle de 0,1 à 20.
12. Procédé suivant la revendication 11, dans lequel la vitesse spatiale horaire de liquide est comprise dans l'intervalle de 0,1 à 5.
13. Procédé suivant l'une quelconque des revendications 1 à 12, dans lequel l'isomérisation est effectuée en présence d'hydrogène.
14. Procédé suivant l'une quelconque des revendications 1 à 13, comprenant en outre l'hydrotraitemen⁶ de l'huile lubrifiante isomérisée.
15. Procédé suivant la revendication 14, dans lequel l'hydrotraitemen¹⁰ est effectuée à une température de 190 °C à 340 °C et sous une pression de 2,9 MPa à 20,8 MPa (400 psig à 3000 psig)
16. Procédé suivant la revendication 14 ou 15, dans lequel l'hydrotraitemen¹⁵ est effectué en présence d'un catalyseur métallique hydrogéné.
17. Procédé suivant l'une quelconque des revendications 1 à 16, dans lequel la charge paraffinique a une teneur en azote organique inférieure à 50 ppm en poids.
18. Procédé suivant la revendication 17, dans lequel la teneur en azote organique est inférieure à 10 ppm en poids.
19. Procédé de production d'une huile lubrifiante ayant un indice de viscosité supérieur à 120 et un point d'écoulement inférieur à -9,4 °C (15 °F), procédé qui comprend le déparaffinage d'une charge paraffinique contenant au moins 50 % de paraffines ou au moins 70 % de carbone paraffinique par isomérisation d'une charge paraffinique sur un catalyseur comprenant (1) un tamis moléculaire non zéolitique à diamètre intermédiaire des pores contenant des motifs d'oxydes tétrahédriques AlO₂ et PO₂ et (2) au moins un métal du Groupe VIII sous une pression de 0,21 MPa à 13,9 MPa (15 psig à 2000 psig).
20. Procédé suivant la revendication 19, dans lequel la charge paraffinique, contenant au moins 50 % de paraffines ou au moins 70 % de carbone paraffinique, est choisie entre un gasoil, une huile lubrifiante de base, une huile synthétique, une huile de ressusage, une paraffine non déshuilee et une paraffine déshuilee.
21. Procédé suivant la revendication 19 ou 20, dans lequel la vitesse spatiale horaire de liquide au cours de l'isomérisation est comprise dans l'intervalle de 0,1 à 20.
22. Procédé suivant l'une quelconque des revendications 19 à 21, comprenant en outre l'hydrotraitemen⁴⁰ de l'huile lubrifiante isomérisée.
23. Huile lubrifiante préparée suivant l'une quelconque des revendications 1 à 22, ayant un indice de viscosité d'au moins 140 et un point d'écoulement non supérieur à -12 °C.
24. Huile lubrifiante préparée suivant l'une quelconque des revendications 1 à 22, ayant un indice de viscosité d'au moins 120 et un point d'écoulement non supérieur à -40 °C.

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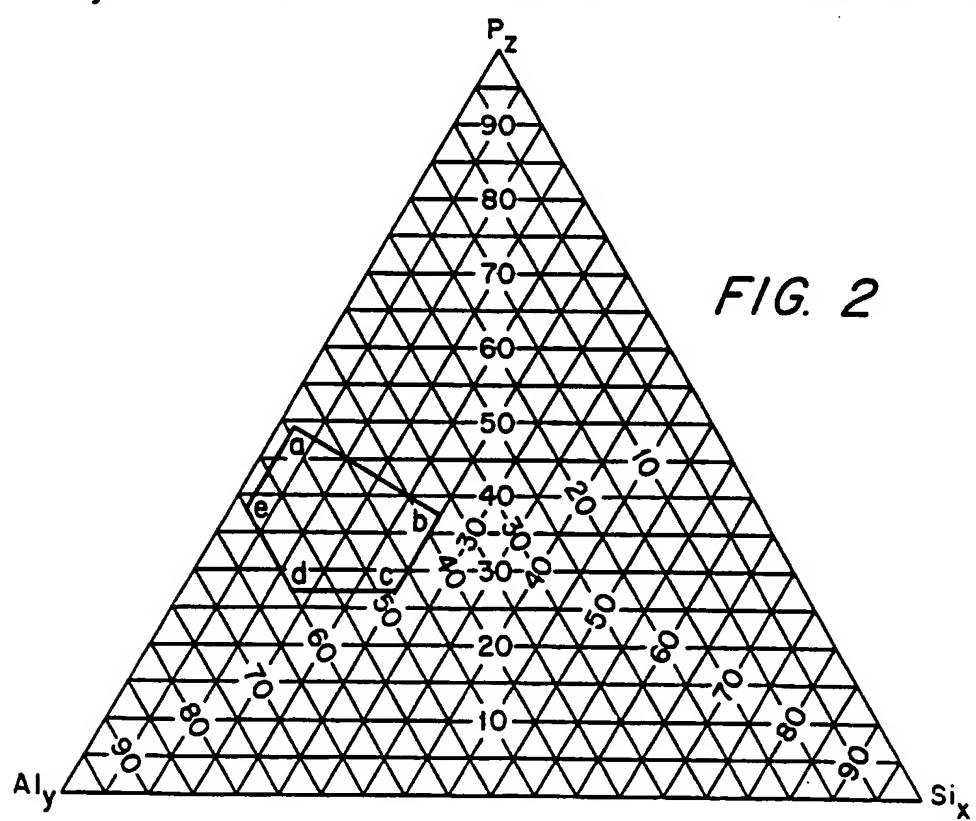
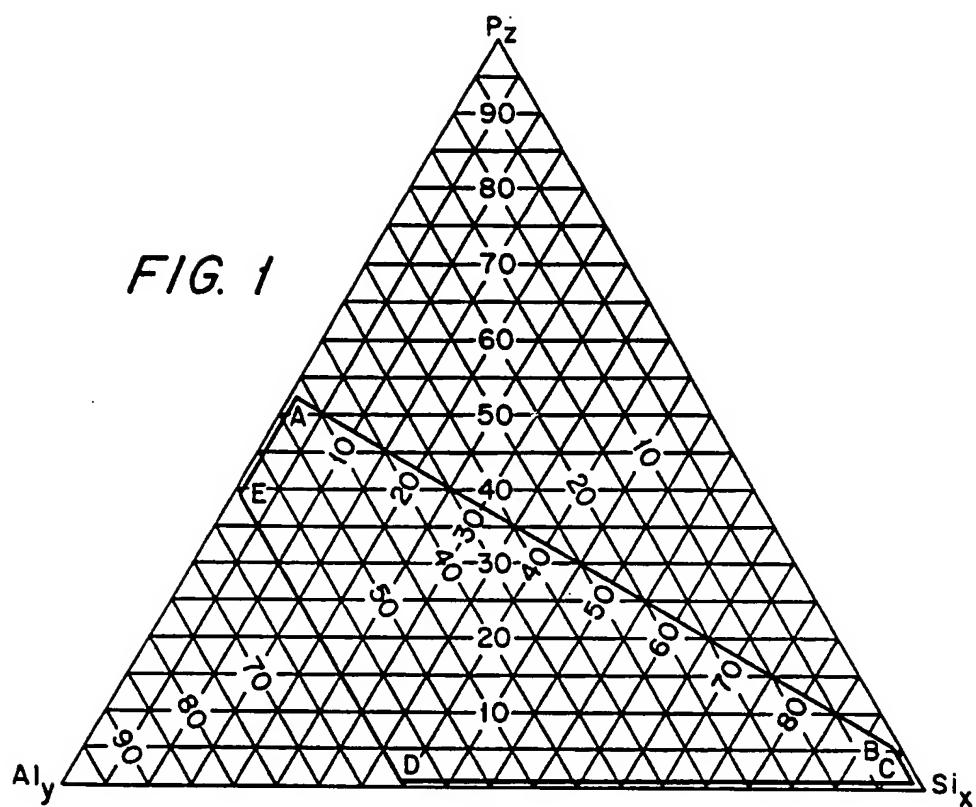


FIG. 3

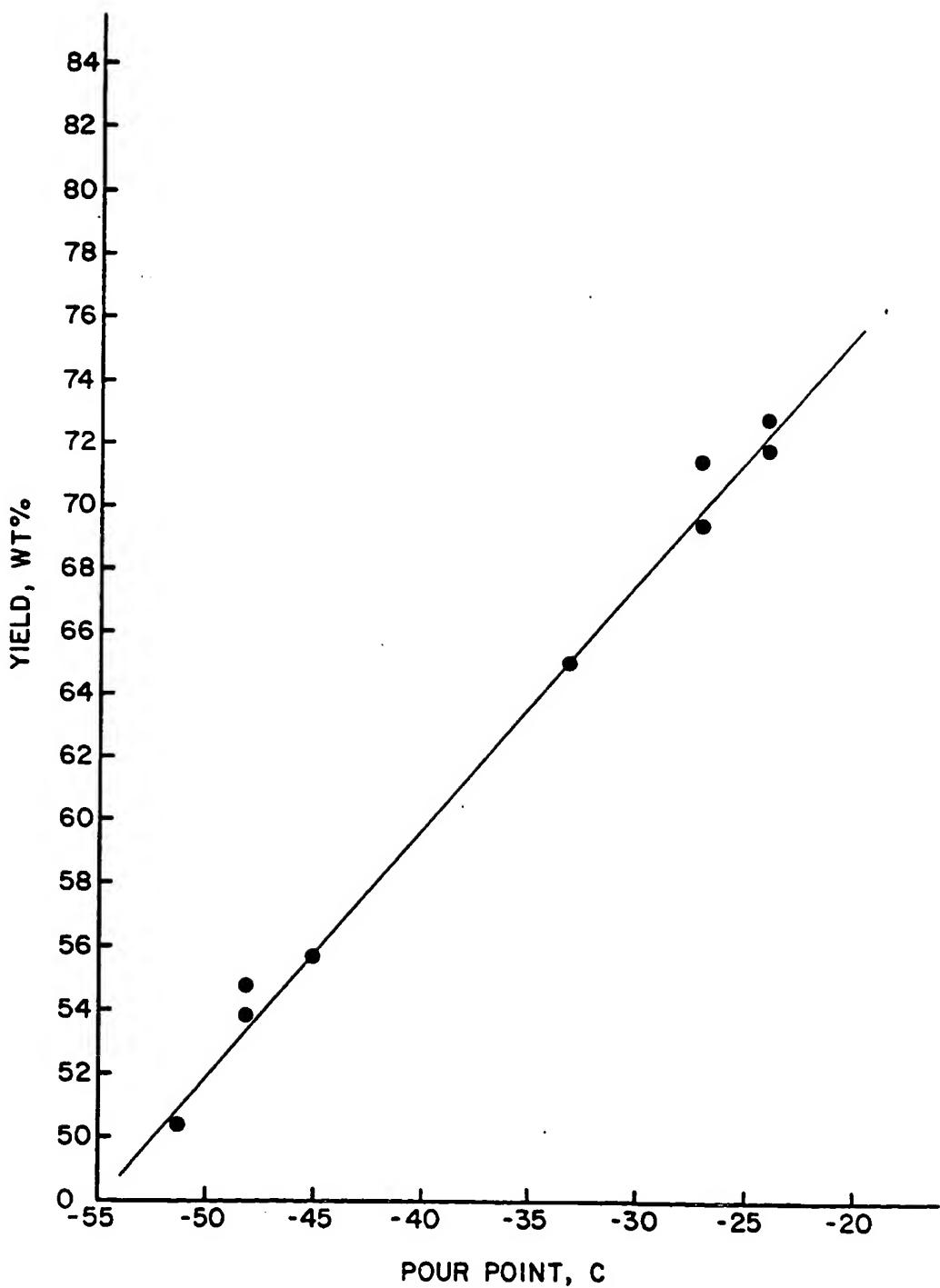
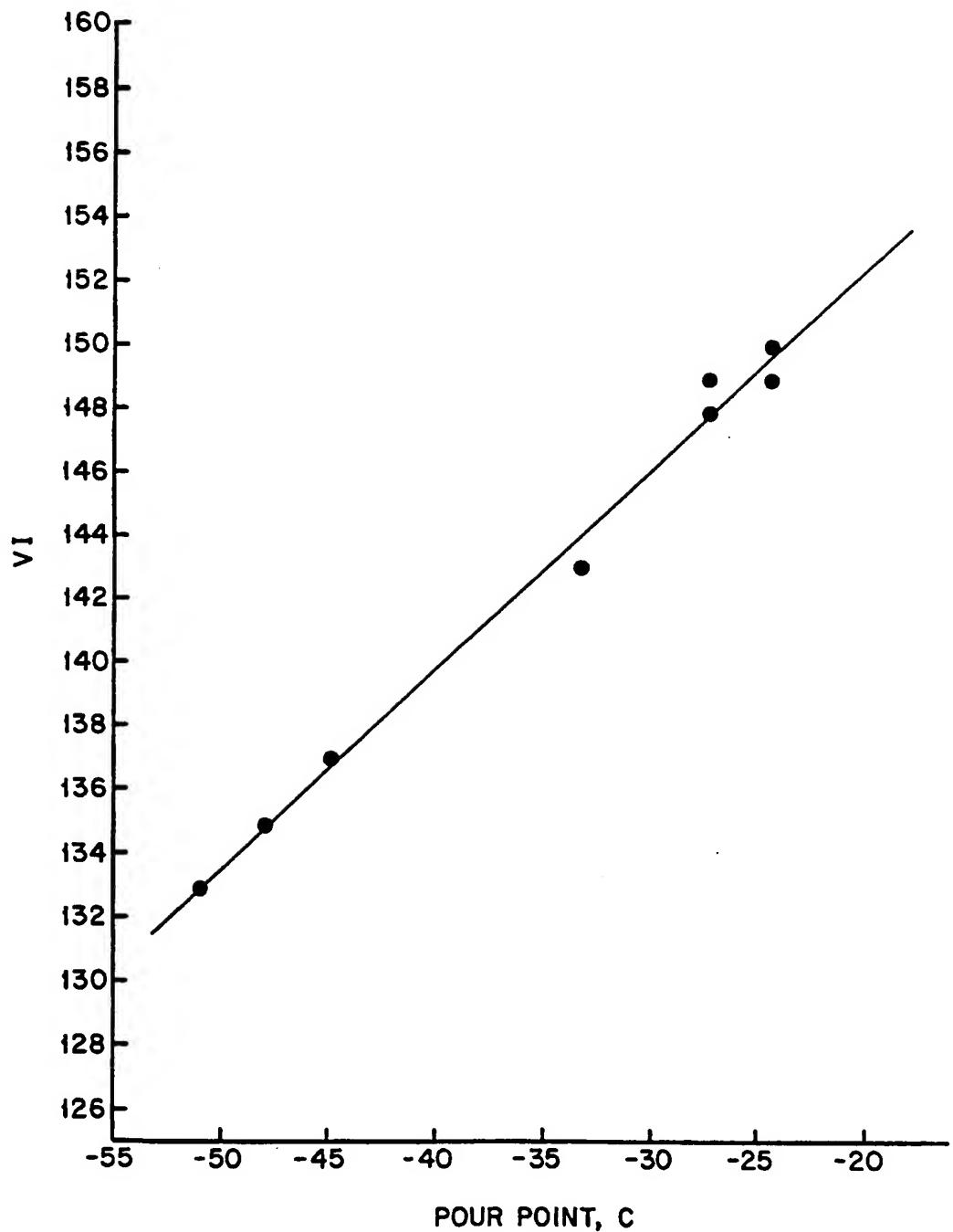


FIG. 4



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